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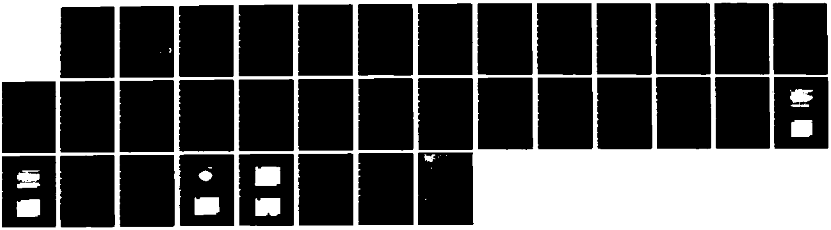

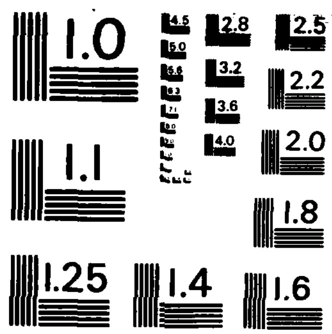

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Uniaxially stretched Nylon-11 films (obtained by melt-pressing and quenching in ice-water, were poled up to 900 KV/cm at 75°C for 30 min. Both piezoelectric strain constant, d_{31}, and piezoelectric stress constant, e_{31}, were found to increase with poling fields. The values reach a plateau at fields of 800 KV/cm. Under identical poling conditions (500 KV/cm; 75°C) d_{31} was found to be ~25% greater than that observed for unoriented films in previous studies while the value for e_{31} was found to be ~85% greater than that for the unoriented films. Static switching

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20. ABSTRACT (continued) d sub 31 e sub 31

experiments were conducted for uniaxially stretched Nylon 11 films, initially prepared by quenching or slow cooling from the melt. Both films exhibited ferroelectric hysteresis behavior in d_{31} and e_{31} indicating that both the quenched and slow cooled points of Nylon-11 are ferroelectric. X-ray diffraction studies for the plasticized and uniaxially stretched slow cooled films indicate that the crystallites may be reorienting in a way consistent with dipole alignment in field direction.

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Technical Report No. 4

PIEZOELECTRIC PROPERTIES AND FERROELECTRIC HYSTERESIS EFFECTS IN UNAXIALLY STRETCHED NYLON-11 FILMS

by

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I. INTRODUCTION

Several studies have been made with respect to field-induced crystal transitions[1-5,18], time dependence of film polarization[6-8], and dependence of the piezoelectric activity on the field-induced changes in the crystalline regions of Poly(vinylidene fluoride) films. Ferroelectric hysteresis effects have also been studied. Conventional ac techniques have been used to observe the hysteresis loop[9] between the electric displacement and applied electric field for stretched films of PVDF with the form I crystalline phase. Biaxially oriented PVF_2 films, subjected to a sequence of rectangular voltage steps[10] of successive opposite polarities, produce P-E hysteresis loops and switching currents typical of ferroelectrics. Switching times of several minutes were observed. Tamura[11] et al. used an electric field of 970 KV/cm (at 50 Hz) and observed the hysteresis characteristic between the field E and the polarization P at room temperature for uniaxially stretched PVF_2 and reported switching times of the order of 10 m sec. for polarization reversal. Infrared transmission[8] techniques have been used to show the hysteresis like behaviour of CF_2 dipoles in the crystalline regions of biaxially oriented PVF_2, when the field is cycled between positive and negative values.

Static switching experiments may also be performed and have the advantage that they may take into account the long time dependence of polarization development. In addition, since these measurements are not made under field, they exclude contributions from film conductivity and from the amorphous regions . Biaxially oriented PVF_2 films[12] subjected to a series of static positive and negative poling fields produce hysteresis curves for the piezoelectric strain constant, d_{31}, and

the piezoelectric stress constant, e_{31}, showing the ferroelectric characteristics of phase IV (polar phase II). X-ray measurements show a field-induced phase transition from phase II to phase IV. It is suggested from these X-ray studies taken during the static switching studies, that reversal of polarization occurs in two steps: a transformation back to phase II, followed by another transformation to phase IV. The contribution to the polarization from phase I crystals also exhibits cyclic behaviour and two switching mechanisms have been proposed: for low fields, 180° flipping, while for higher fields, 60° flipping.

Investigations have also been made of the piezoelectric activity in poled Nylon-11 films. The response is higher[13] for poled quenched films as compared to the melt crystallized films under identical poling conditions. The melt crystallized material is termed the α' form here as it exhibits small d spacing differences from those reported for the α-form (reference to Kawaguchi). This will be discussed in a separate publication.[14] No detailed studies have been made regarding field-induced crystal transitions for Nylon-11. Studies using X-ray diffraction[15] for unpoled and poled quenched Nylon-11 films indicate that there is no phase transition to the α or α' phases during poling. This suggests the existence of a polar phase in the quenched films. This will be discussed in a separate publication.[14] In other preliminary studies, uniaxially drawn Nylon-11 quenched films[16] showed higher piezoelectric activity when compared to unoriented films under identical poling conditions. In a recent study of electric-field induced changes in the X-ray diffraction pattern[17] of Nylon-11 films which the authors state to be a mixture of the α' and γ crystal phases, it was observed that the crystallites orient in such a way that the dipoles are preferentially aligned in the direction of the poling field. A correlation between the piezoelectric response and

the degree of orientation observed in the X-ray scans was observed.

In order to obtain further information about the effects of polarization and possibility of crystal phase transitions, we studied the poling field dependence of the piezoelectric behaviour for uniaxially stretched Nylon-11 quenched films. For the second part of the study, static switching experiments were used to determine if Nylon 11 is a ferroelectric and if so, to study the polarization reversibility for the two types of films: uniaxially stretched Nylon-11 quenched films, and plasticized and uniaxially stretched Nylon-11 (α') films. The reasons for plasticization in the latter case was to be able to stretch the films at lower temperatures and to increase their piezoelectric activity. X-ray diffraction and differential scanning calorimetry were used to study the crystalline regions after various poling histories.

II. EXPERIMENTAL

Nylon-11 films were made in two different forms. Melt-pressing at 205-210°C followed by slow cooling gave the α' form. The quenched films were obtained by melt-pressing the Nylon-11 powder in the same temperature range and then quenching the molten films into ice water. In both cases, the Nylon-11 powder was pressed between aluminum foil at 3000 psi. The quenched films were then uniaxially stretched to a draw-ratio of 2.5:1 in an Instron at 23°C at an extension rate of 0.05"/min. The α' films were very difficult to stretch at temperatures below 120°C, so we decided to plasticize these films before stretching. The films were plasticized by immersing in 2-Ethyl 1,3 Hexanediol at 75°C for 1/2 hour. The content

of plasticizer was found to be 11.6% by weight. These films were then uniaxially stretched to a draw-ratio of 2.4:1 at 50°C at the same extension rate as before. Flat-film X-ray patterns and wide angle diffraction scans were taken in both cases.

Rectangular samples were cut from the stretched films, keeping the longer dimension parallel to the draw-direction. Electrodes were coated on the samples using silver-paint. Poling was carried out using a conventional two-terminal method with the sample placed between two copper electrodes. An automatic voltage ramper raised the voltage across the sample to the desired level at a constant rate. After reaching the desired field, the temperature was raised to the poling temperature. On completion of poling, the temperature was lowered with the field on. The quenched films were poled at 75°C for 30 minutes. Because of excessive breakdown at 75°C, the α' samples were poled at 50°C for the same length of time. All samples were poled in a vacuum of $\sim 10^{-5}$ torr. Uniaxially stretched quenched films were subjected to poling fields up to 900 KV/cm. The Piezoelectric strain constant, d_{31}, piezoelectric stress constant, e_{31}, dielectric constant and modulus were measured in a Toyo-Seiki Piezotron at 3 Hz. To study the hysteresis effects, films were taken in static steps up to poling fields of 300 KV/cm and then the poling field direction was reversed. Two cycles were completed with fields up to 350 KV/cm in each direction. The same sample was used for all fields. During the electrical measurements performed after each static poling step, the hard silver-paint electrodes were removed and soft silver-epoxy was used for the electrodes. The α' films were also poled for two cycles, with fields up to 350 KV/cm in each direction of poling.

Flat-film X-ray patterns and wide angle diffraction scans were taken at different stages of poling. Differential scanning calorimetry studies were made using a Perkin-Elmer DSC 1-B and a heating rate of 10°C/min.

III. RESULTS AND DISCUSSIONS

A. Electrical Measurements

Fig. 1(a) and Fig. 1(b) show the results for uniaxially stretched Nylon-11 quenched films. The values for both d_{31} and e_{31} increase with increasing poling fields. The value for d_{31} (3.47 PC/N) is ∿25% greater than that observed for unoriented films in previous studies[13] under identical poling conditions (500 KV/cm; 75°C) while the value for e_{31} (5.47 mC/m2) is ∿85% greater than that for the unoriented films. The values tend to reach a plateau at fields of 800 KV/cm under the given poling conditions. The highest value for d_{31} obtained was 4.2 PC/N and that for e_{31} was 7.2 mC/m^2 at a poling field of 900 KV/cm. The samples could not be poled at higher fields without breakdown occurring. The frequency of dielectric breakdown increased rapidly at fields above 600 KV/cm. The modulus of all the samples was also measured in the Piezotron and the ratio of e_{31}/d_{31} = modulus, matched within experimental error. Fig. 2 shows that the modulus of the samples after poling at various fields remains constant. Fig. 3 shows the dielectric constant at different poling fields. The values are essentially constant up to fields of 700 KV/cm and then show a slight increasing trend.

Fig. 4 shows the hysteresis loop for d_{31} for uniaxially stretched Nylon-11 quenched films. During the first cycle, the sample was initially subjected to poling fields of 100, 200, and 300 KV/cm and the value of d_{31} increased as observed earlier. The dashed line connecting the point

at 300 KV/cm and 0 KV/cm indicates that d_{31} is unchanged if the poling direction is not reversed. Then, the sample was subjected to poling fields in the reverse direction: -100, -200, -300, and -350 KV/cm. The value of d_{31} went through zero at a field of -180 KV/cm. At a poling field of -350 KV/cm, the value of d_{31} was -1.33 PC/N. When the sample was again subjected to forward poling fields, the value of d_{31} went through zero again at a field of 180 KV/cm. The second cycle of reversed and forward poling produced similar paths within experimental error. Fig. 5 shows the hysteresis loop for the piezoelectric stress constant, e_{31}. The behaviour is essentially similar in trend to that of the piezoelectric strain constant.

Fig. 6 and Fig. 7 show the hysteresis loops for d_{31} and e_{31}, respectively, for plasticized, uniaxially stretched Nylon-11 (α') films. When the sample was first subjected to a series of forward poling fields, the values of d_{31} and e_{31} increased sharply. At a poling field of 350 KV/cm, d_{31} was 2.75 PC/N and e_{31} was 1.7 mC/m^2. Again, the dashed line connecting the points at 350 KV/cm and 0 KV/cm only indicates that the value of d_{31} does not change if the poling direction is not reversed. On subsequent reversed poling, both d_{31} and e_{31} go through zero at a field of $\sim$250 KV/cm. At -350 KV/cm, d_{31} was -1.5 PC/N and e_{31} was -1.08 mC/m^2. When the poling direction is switched back to the positive direction, the value again goes through zero but at a field of 180 KV/cm, the value obtained for the quenched films. Similar behaviour is observed during the second cycle; however, the values of d_{31} and e_{31} are much lower at the end of the second cycle; d_{31} was 0.95 PC/N and e_{31} was 0.64 mC/m^2. Since the same sample has been subjected to all the poling fields at 50°C, we might be observing an annealing effect resulting in

increased crystal perfection making poling more difficult and leading to a lowering of the values for d_{31} and e_{31}.

B. X-ray Measurements

Fig 8 and Fig. 9 show the X-ray diffraction scans for unpoled (with the same thermal history as the poled sample) and poled (650 KV/cm; 75°C; 30 min.) uniaxially stretched Nylon-11 quenched films in reflection and transmission modes respectively. Fig. 10 shows the flat-film X-ray pattern for these films in transmission mode (X-ray beam normal to the plane of the film and to the c-axis). Fig. 11 shows the flat-film patterns of the samples in edge-reflection mode (X-ray beam parallel to the plane of the film and perpendicular to the c-axis). There is no significant change observed in the scans before and after poling. Fig. 12 and Fig. 13 show the X-ray diffraction scans for unpoled and poled (through two complete cycles of poling of the ferroelectric hysteresis experiment) in reflection and transmission modes respectively of the uniaxial stretched α' films. The unpoled sample had the same thermal history as the poled sample. The intensity of the (010) reflection has decreased significantly compared to that of the (100) reflection for the poled sample in reflection mode (Fig. 12). The complementary (reverse) effect is seen in the scan taken in transmission mode (Fig. 13). This would be expected if the dipoles align perpendicular to the film during poling. In a recent study[17] for Nylon-11 (α) films, similar X-ray changes were observed after poling and were related to the reorientation of crystallites (under the effect of poling fields) so that the dipoles align in the direction of the applied field. Fig. 14 shows the flat-film X-ray pattern of these samples in transmission mode and Fig. 15 show the flat-film pattern in the edge reflection mode.

C. DSC Measurements

Fig. 16 shows DSC endotherms for the uniaxially stretched quenched films. Equal weights of each sample were used. The peak melting point shows an increasing trend with higher poling fields. The melting peak for the unpoled sample is 192.5°C, while for the sample poled at 800 KV/cm, it has increased to 194°C. The increase may be attributed to the fact that annealing is occurring under high poling fields.

Fig. 17 shows the DSC melting endotherms for the uniaxially stretched, plasticized Nylon-11 (α') films. For the unpoled sample (thermal history the same as for the poled sample) the melting peak occurs at 187.5°C, while for the poled sample (at the end of the hysteresis cycles) the peak is at 190°C. Premelting is indicated by the slope in the base line observed before the melting endotherm of the poled sample.

IV. CONCLUSION

Previous studies have shown crystal phase transitions and reorientation taking place during poling of PVF_2 films. Ferroelectric hysteresis studies have shown that the direction of polarization can be reversed when fields of sufficient magnitudes are applied. The increase in piezoelectric response with higher poling fields has also been related to field-induced crystal phase transitions in PVF_2 films. The present study shows an increase in the piezoelectric response for Nylon-11 films (uniaxially stretched) with increase in the poling fields. Nylon-11 quenched films show appreciably higher piezoelectric activity in the oriented form as compared to the unoriented form studied earlier.[13] Under identical poling conditions, the value of d_{31} was found to be 25% higher for the uniaxially stretched

quenched films compared to the unoriented films. The values for both d_{31} and e_{31} reach saturation at fields nearing 800 KV/cm.

The static switching experiments have shown that both the quenched and α' form Nylon-11 films are ferroelectric as indicated by the hysteresis behaviour of the piezoelectric strain constant and the piezoelectric stress constant.

X-ray studies show that there is no crystal phase transition occurring during poling of these films. The crystallites seem to re-orient in a way consistent with dipole alignment in the field direction as indicated by the X-ray studies for the uniaxially stretched, plasticized α' films. In previous studies[17], a similar field-induced reorientation was observed for unoriented α' films. DSC studies indicate that annealing may be taking place under high poling fields resulting in an increase in melting point with increasing poling field.

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FIGURE CAPTIONS

1(a). Piezoelectric strain constant, d_{31}, vs. poling fields for uniaxially stretched Nylon 11 (quenched) films; poled at 75°C for 30 min.

1(b). Piezoelectric stress constant, e_{31}, vs. poling fields for uniaxially stretched Nylon 11 (quenched) films, poled at 75°C for 30 min.

2. Modulus at different poling fields for uniaxially stretched Nylon 11 (quenched) films.

3. Dielectric constant vs. poling field for uniaxially stretched Nylon 11 (quenched) films.

4. Hysteresis loop of d_{31} for uniaxially stretched Nylon 11 (quenched) films.

5. Hysteresis loop of e_{31} for uniaxially stretched Nylon 11 (quenched) films.

6. Hysteresis loop of d_{31} for uniaxially stretched, plasticized Nylon 11 (α') films.

7. Hysteresis loop of e_{31} for uniaxially stretched, plasticized Nylon 11 (α') films.

8. X-ray diffraction scans (reflection mode) for unpoled and poled uniaxially stretched Nylon 11 (quenched) films.

9. X-ray diffraction scans (transmission mode) for unpoled and poled uniaxially stretched Nylon 11 (quenched) films.

10. Flat film X-ray patterns for unpoled and poled Nylon 11 (quenched) films; X-ray beam normal to the plane of the film and the c-axis.

11. Flat-film X-ray pattern for unpoled and poled Nylon 11 (quenched) films; X-ray beam parallel to the plane of the film and perpendicular to the c-axis.

12. X-ray diffraction scans (reflection mode) for unpoled and poled, uniaxially stretched, plasticized Nylon 11 (α') films.

13. X-ray diffraction scans (transmission mode) for unpoled and poled, uniaxially stretched, plasticized Nylon 11 (α') films.

14. Flat-film X-ray patterns for unpoled and poled, uniaxially stretched, plasticized Nylon 11(α') films; X-ray beam normal to the plane of the film and perpendicular to the c-axis.

15. Flat-film X-ray patterns for unpoled and poled, uniaxially, plasticized Nylon 11(α') films; X-ray beam parallel to the plane of the film and perpendicular to c-axis.

16. DSC endotherms for unpoled and poled, uniaxially stretcned Nylon 11 (quenched) films.

17. DSC endotherms for unpoled and poled, uniaxially stretched, plasticized Nylon 11 (α') films.

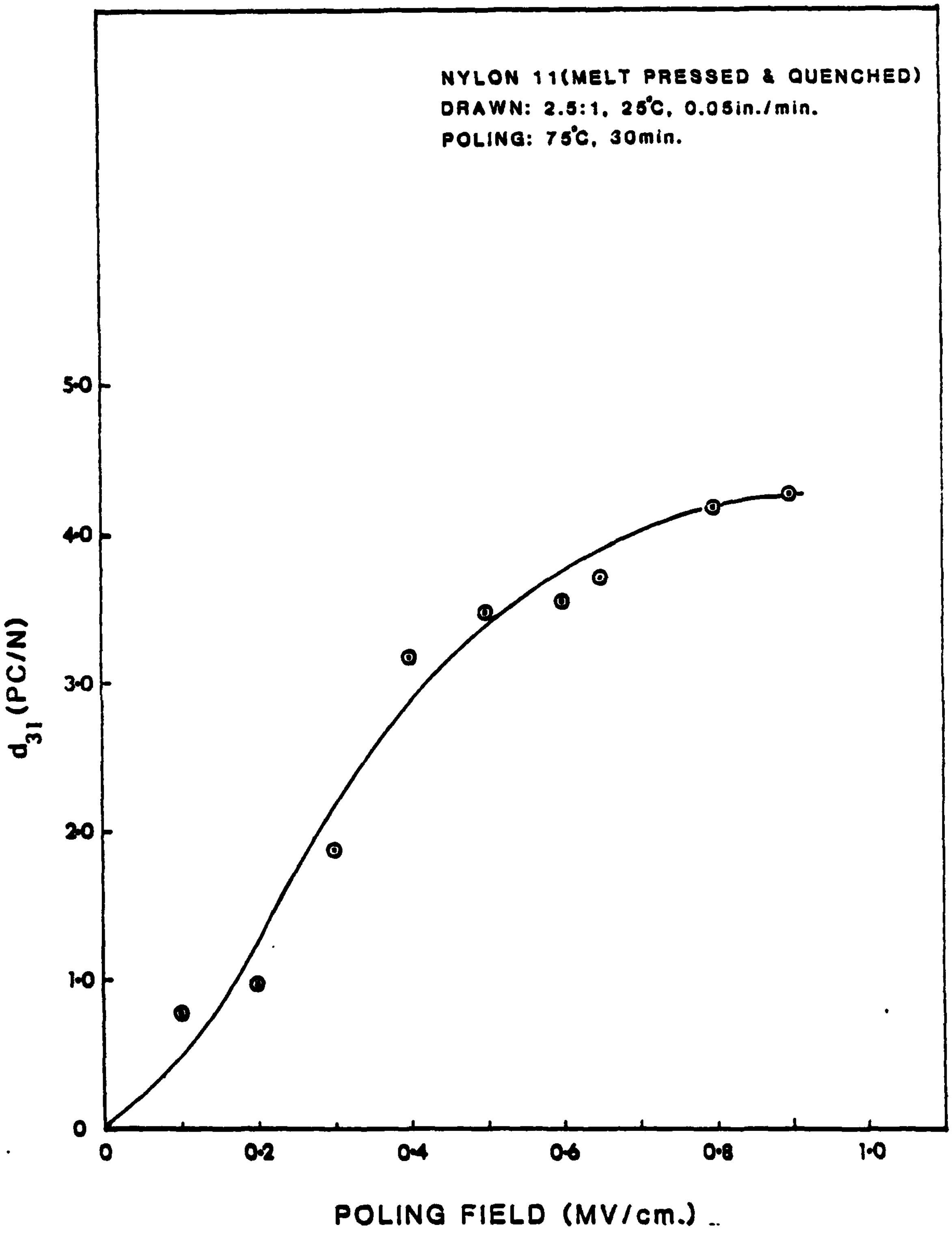

NYLON 11(MELT PRESSED & QUENCHED)
DRAWN: 2.5:1, 25°C, 0.05in./min.
POLING: 75°C, 30min.
d_{31} (PC/N)
5·0
4·0
3·0
2·0
1·0
0
0
0·2
0·4
0·6
0·8
1·0
POLING FIELD (MV/cm.)

Fig. 1(a)

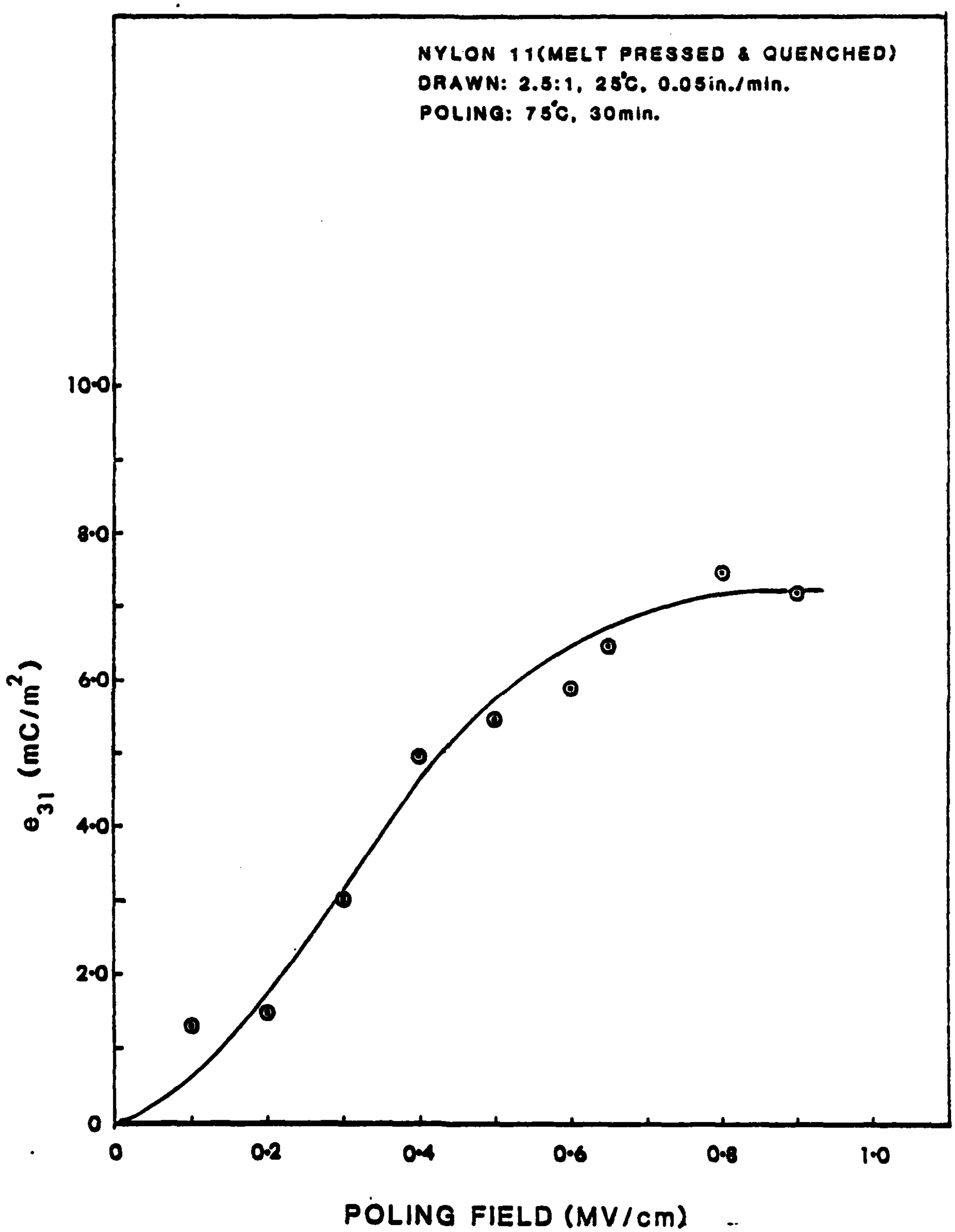

NYLON 11(MELT PRESSED & QUENCHED)
DRAWN: 2.5:1, 25°C, 0.05in./min.
POLING: 75°C, 30min.
10·0
8·0
6·0
4·0
2·0
0
e_{31} (mC/m^2)
0
0·2
0·4
0·6
0·8
1·0
POLING FIELD (MV/cm)

Fig. 1(b)

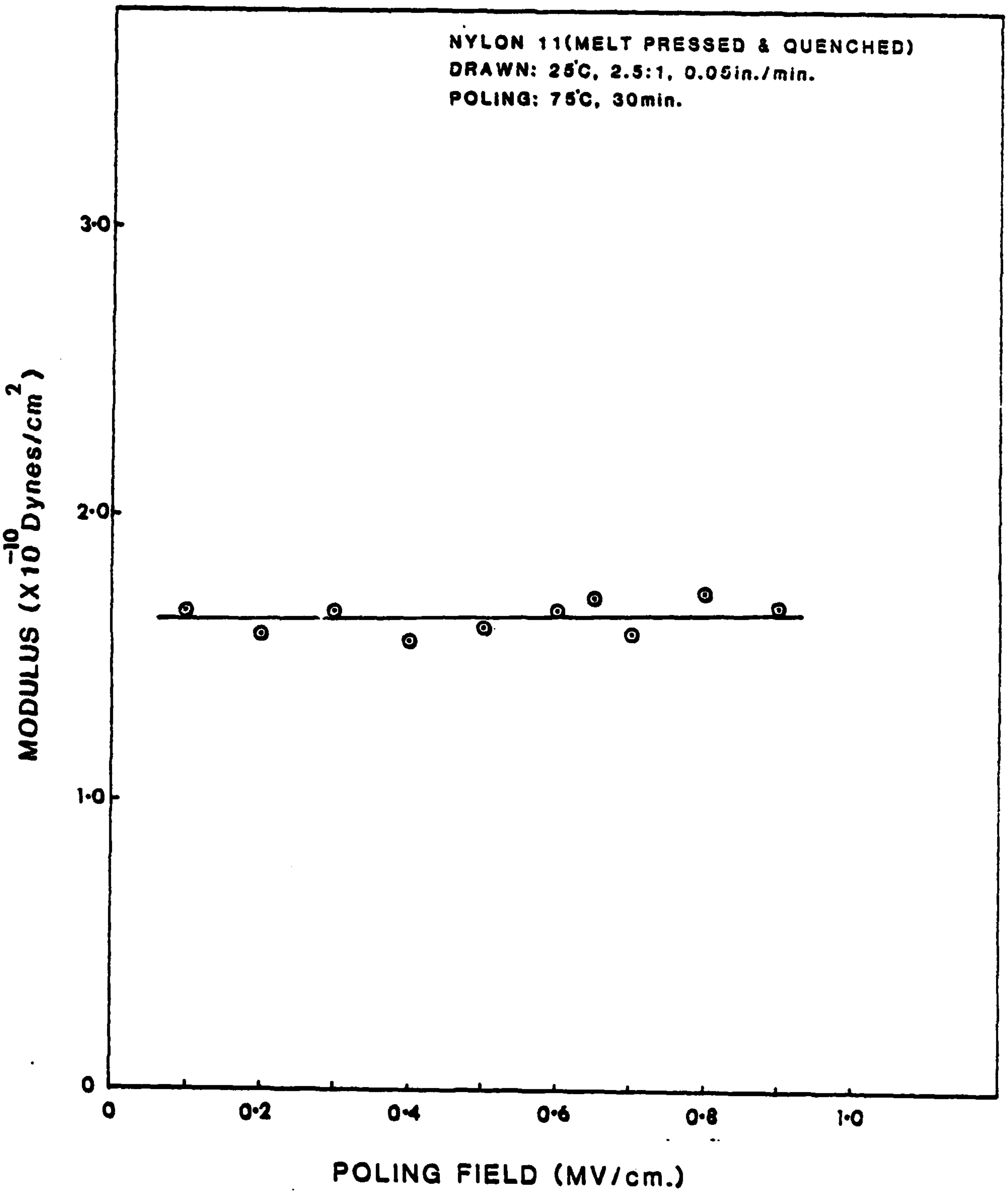
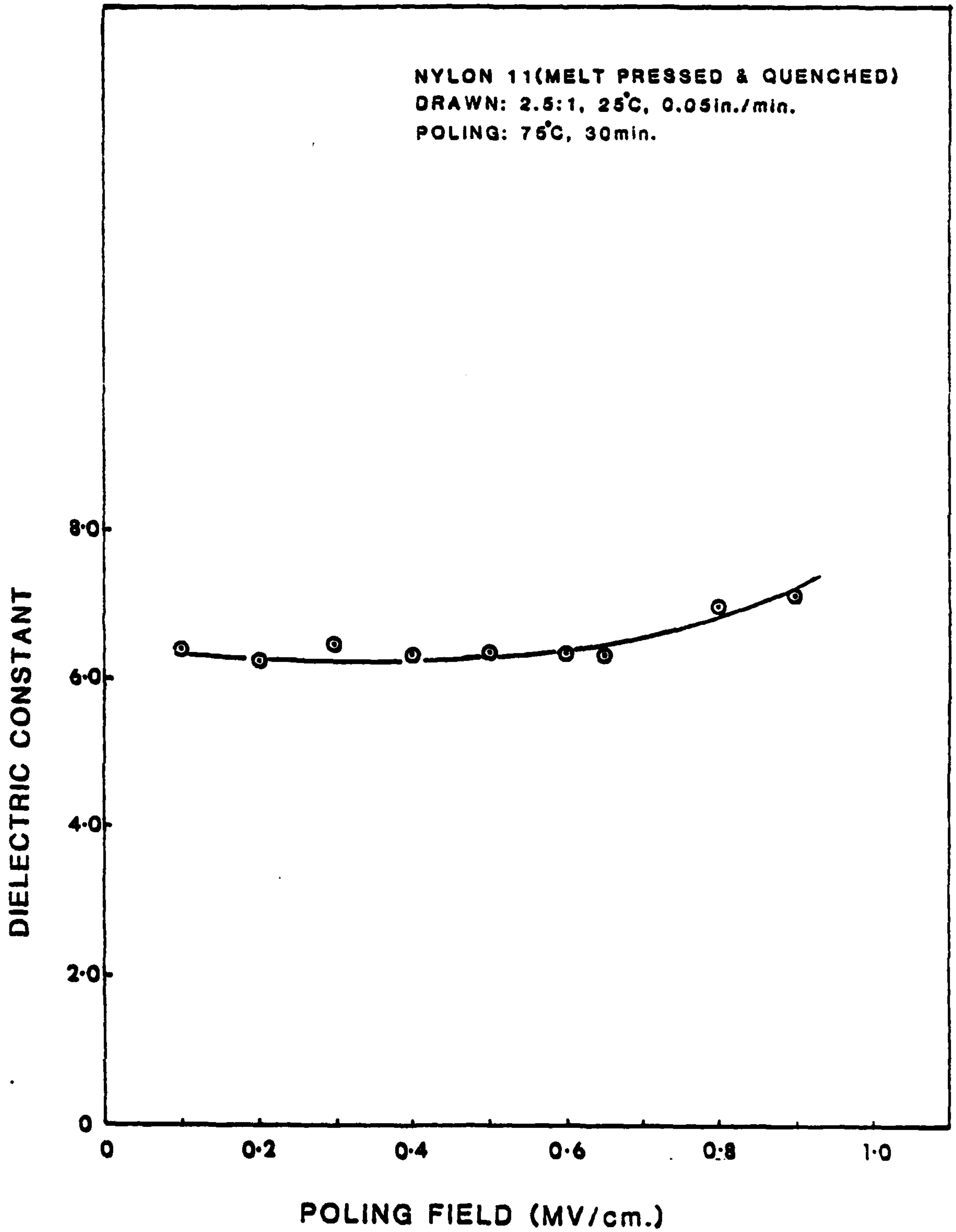

NYLON 11(MELT PRESSED & QUENCHED)
DRAWN: 25°C, 2.5:1, 0.05in./min.
POLING: 75°C, 30min.
MODULUS (X10^{-10} Dynes/cm^2)
3·0
2·0
1·0
0
0
0·2
0·4
0·6
0·8
1·0
POLING FIELD (MV/cm.)

Fig. 2



NYLON 11(MELT PRESSED & QUENCHED)
DRAWN: 2.5:1, 25°C, 0.05in./min.
POLING: 75°C, 30min.
DIELECTRIC CONSTANT
8·0
6·0
4·0
2·0
0
0
0·2
0·4
0·6
0·8
1·0
POLING FIELD (MV/cm.)

Fig. 3

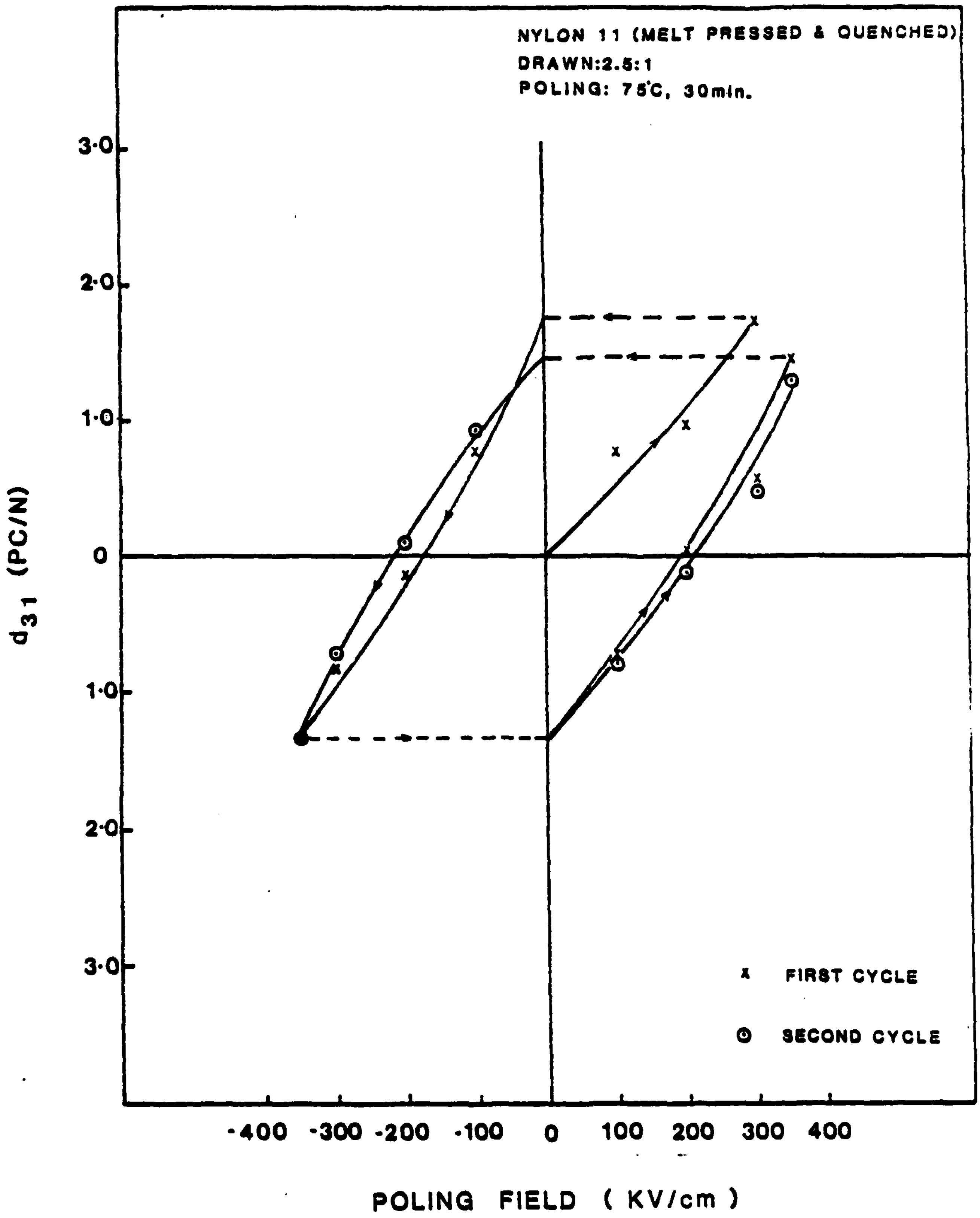
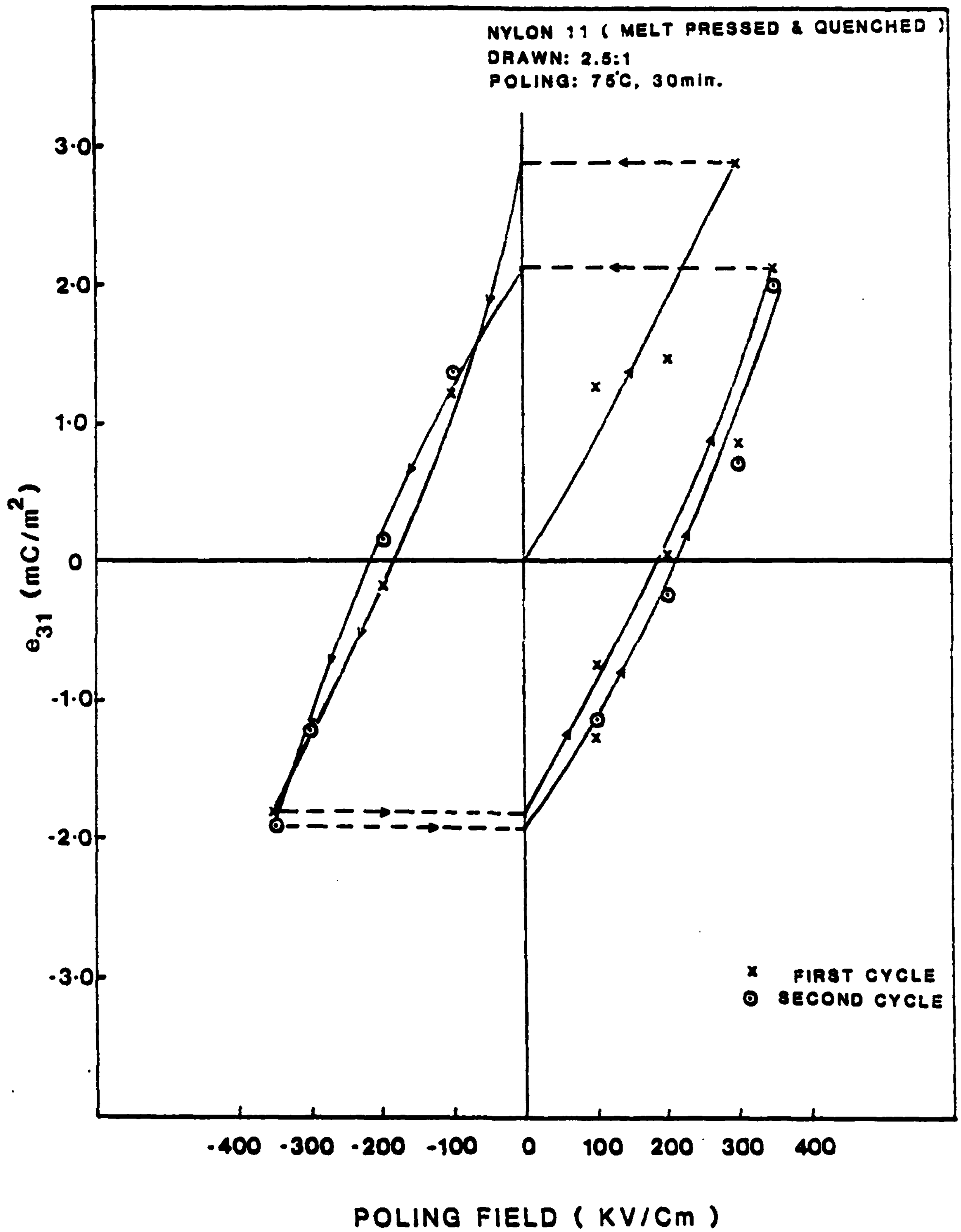

NYLON 11 (MELT PRESSED & QUENCHED)
DRAWN:2.5:1
POLING: 75°C, 30min.
3·0
2·0
1·0
0
1·0
2·0
3·0
d_{31} (PC/N)
FIRST CYCLE
SECOND CYCLE
-400 -300 -200 -100 0 100 200 300 400
POLING FIELD (KV/cm)

Fig. 4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DRAWN: 2.5:1
POLING: 75°C, 30min.
3·0
2·0
1·0
0
-1·0
-2·0
-3·0
e_{31} (mC/m^2)
× FIRST CYCLE
⊙ SECOND CYCLE
-400 -300 -200 -100 0 100 200 300 400
POLING FIELD (KV/Cm)

Fig. 5

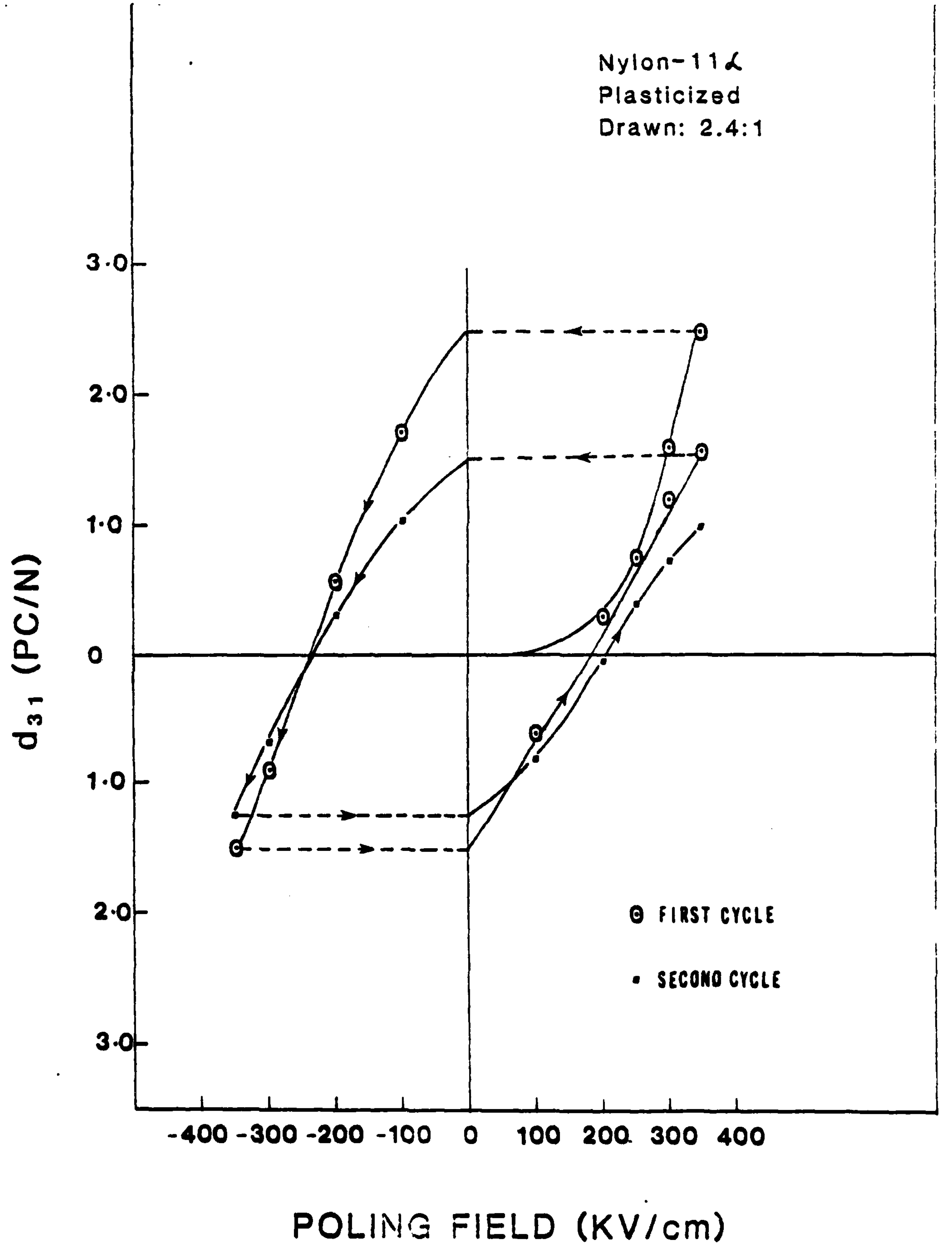

Nylon-11α
Plasticized
Drawn: 2.4:1
3·0
2·0
1·0
0
1·0
2·0
3·0
d_{31} (PC/N)
-400 -300 -200 -100 0 100 200 300 400
POLING FIELD (KV/cm)
FIRST CYCLE
SECOND CYCLE

Fig. 6

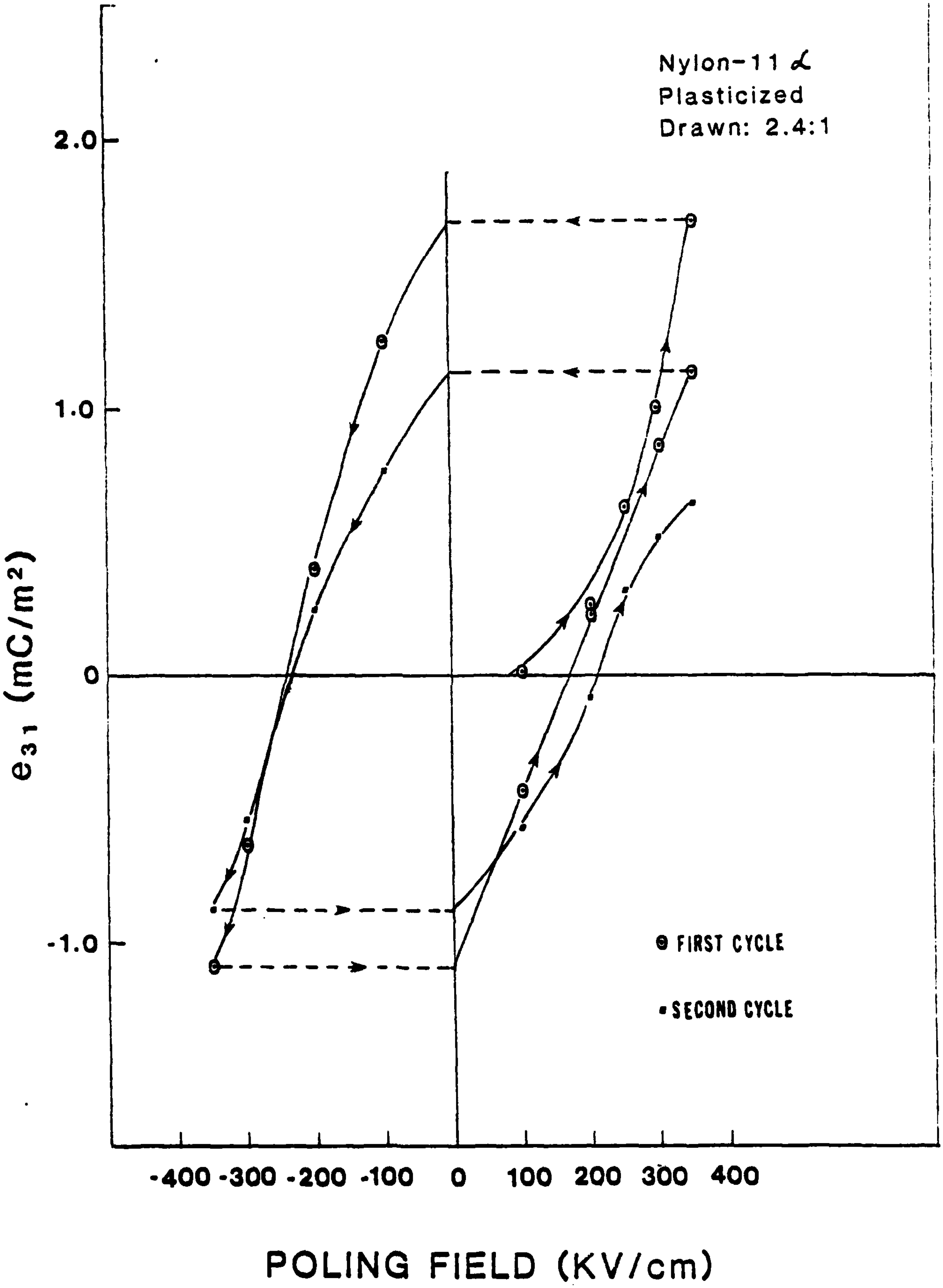
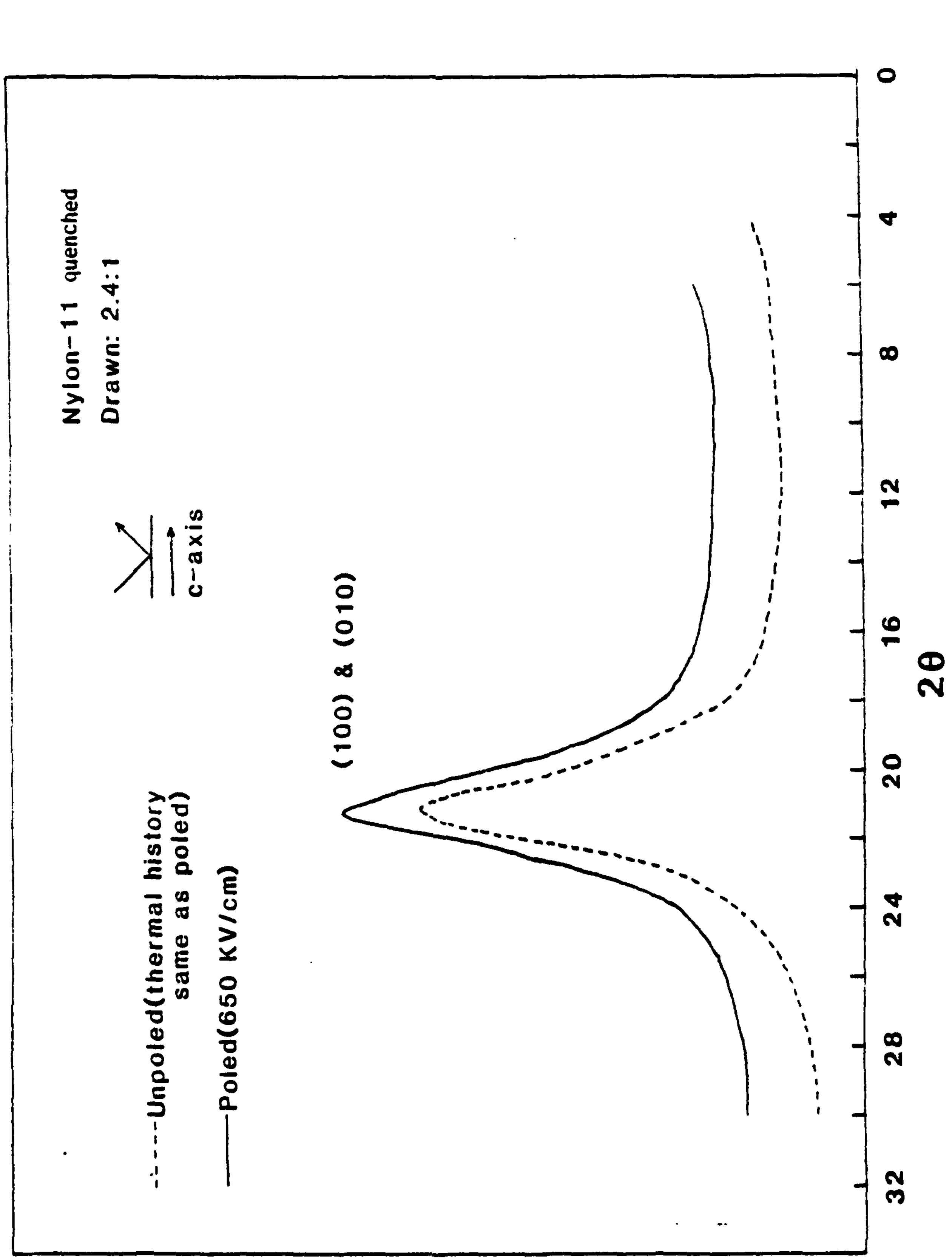
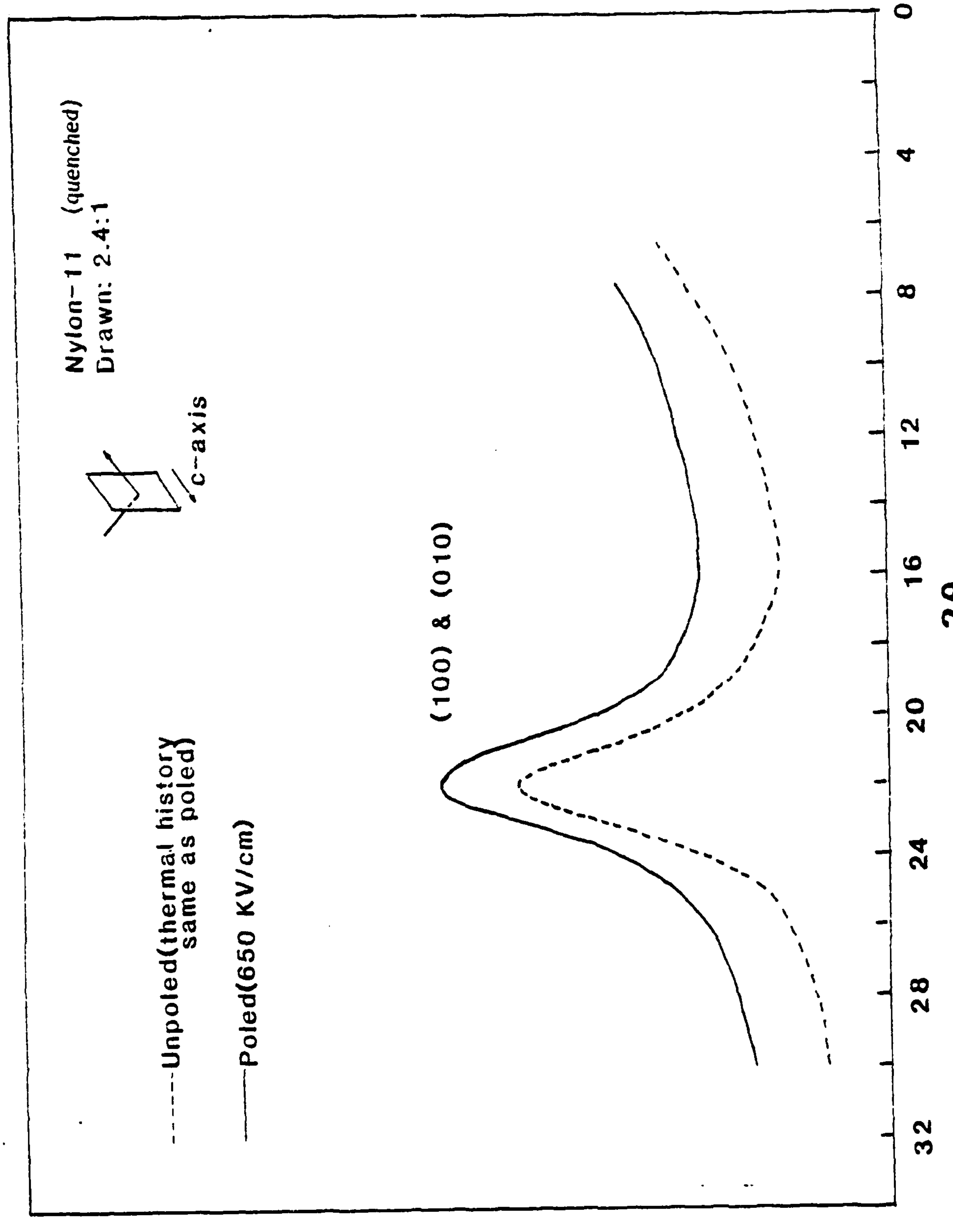

Nylon-11 α
Plasticized
Drawn: 2.4:1
2.0
1.0
0
-1.0
e_{31} (mC/m²)
FIRST CYCLE
SECOND CYCLE
-400 -300 -200 -100 0 100 200 300 400
POLING FIELD (KV/cm)

Fig. 7



Nylon-11 quenched
Drawn: 2.4:1
c-axis
(100) & (010)
Unpoled(thermal history same as poled)
Poled(650 KV/cm)
0
4
8
12
16
20
24
28
32
2θ

Fig. 8



Nylon-11 (quenched)
Drawn: 2.4:1
c-axis
(100) & (010)
----Unpoled(thermal history same as poled)
——Poled(650 KV/cm)
0
4
8
12
16
20
24
28
32
2θ

Fig. 9

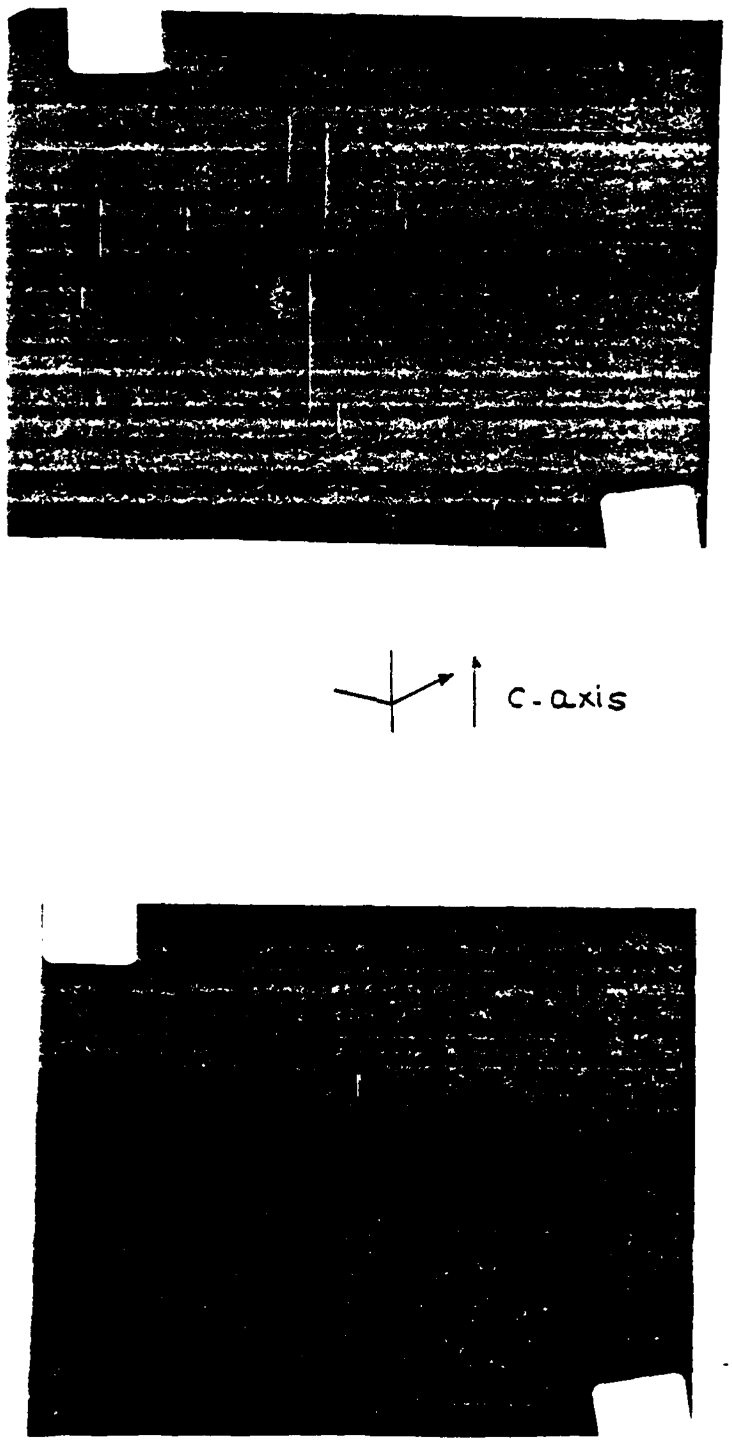


Fig. 10

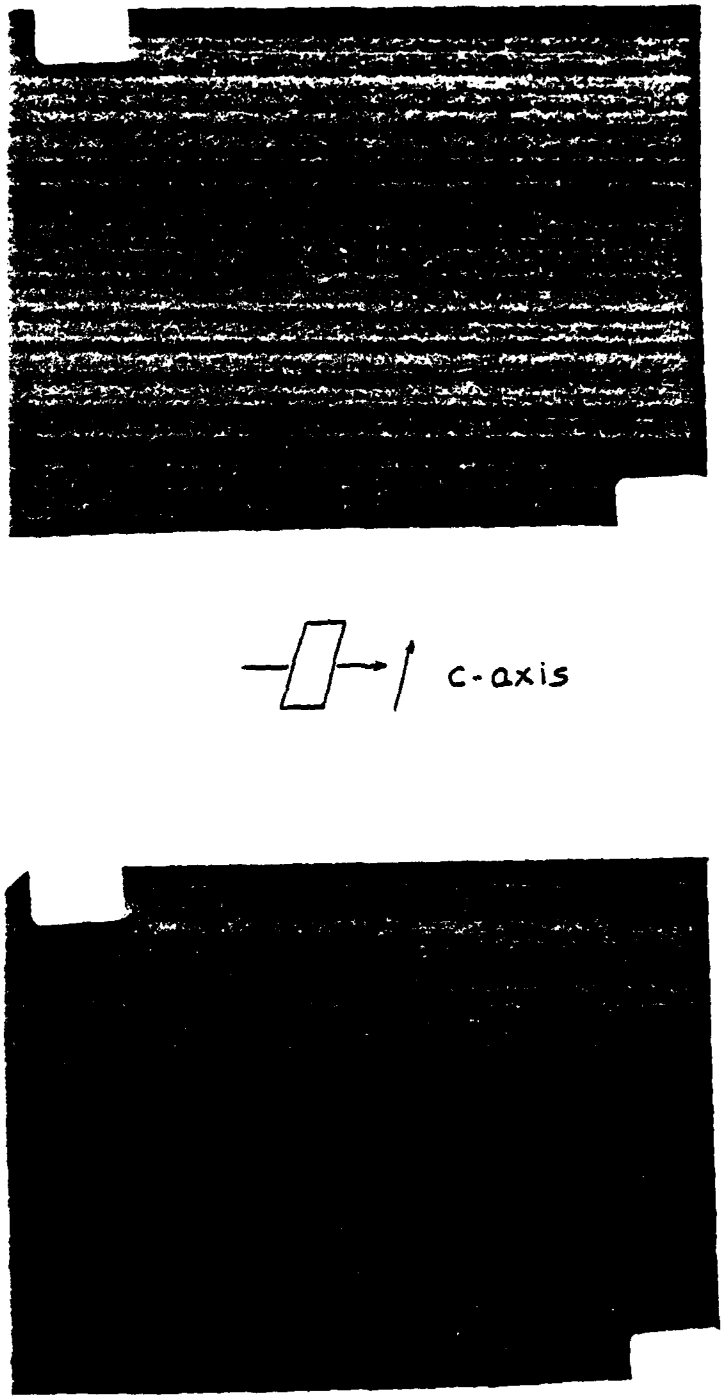
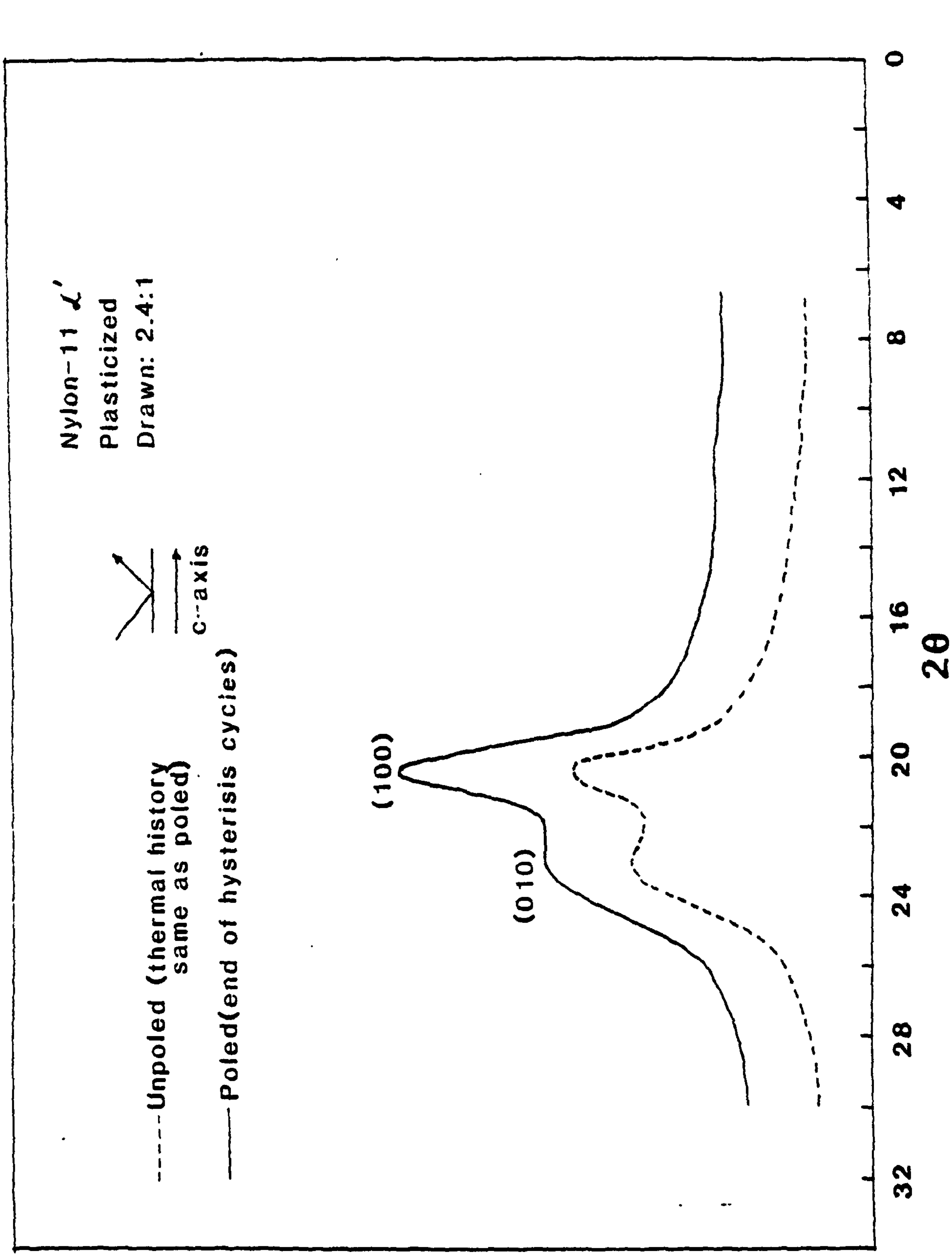


Fig. 11



Nylon-11 δ'
Plasticized
Drawn: 2.4:1
c-axis
----Unpoled (thermal history same as poled)
——Poled(end of hysterisis cycles)
(100)
(010)
0
4
8
12
16
20
24
28
32
2θ

Fig. 12

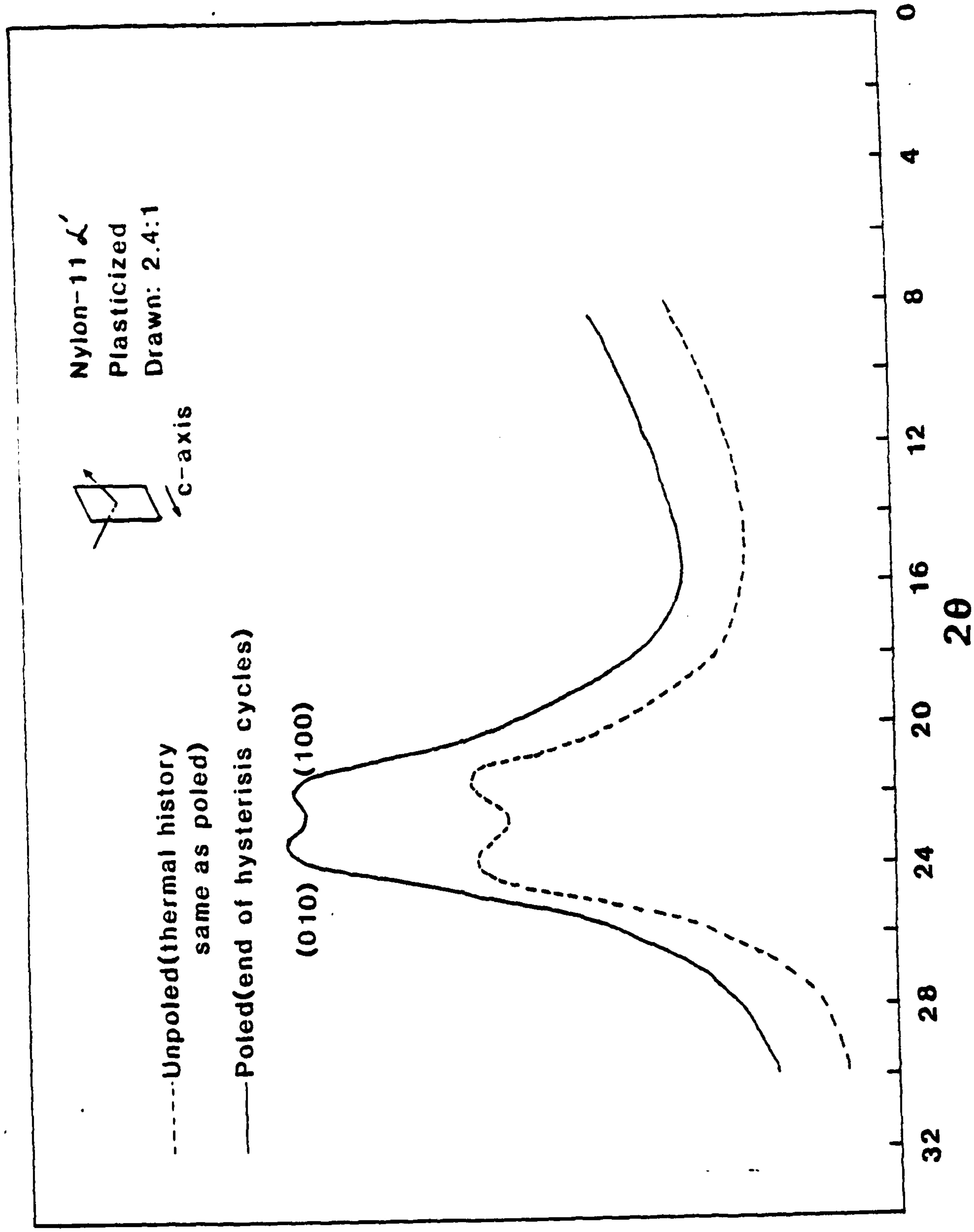

Nylon-11 α'
Plasticized
Drawn: 2.4:1
c-axis
Unpoled(thermal history same as poled)
Poled(end of hysterisis cycles)
(010)
(100)
32
28
24
20
16
12
8
4
0
2θ

Fig. 13

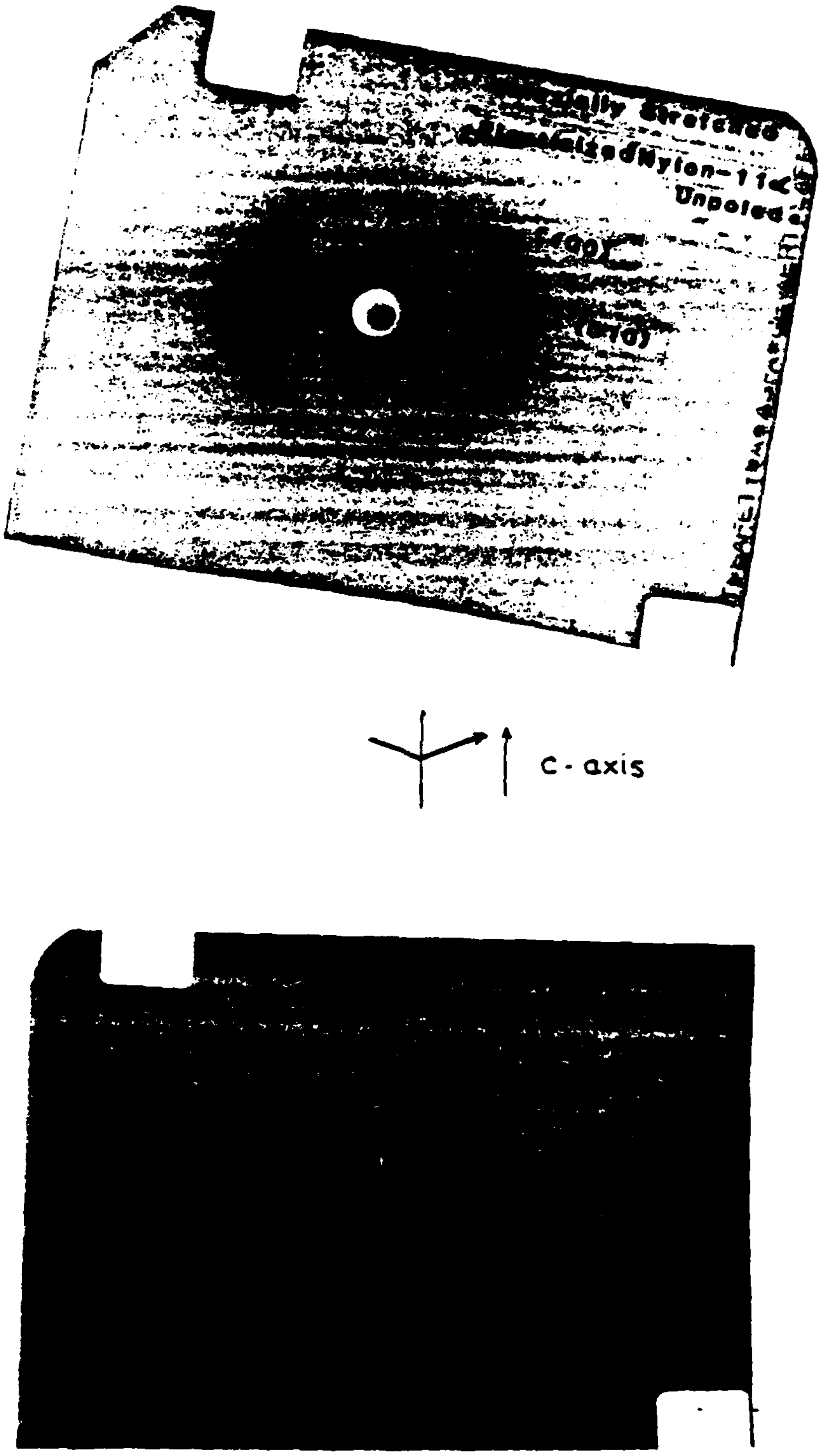


Fig. 14

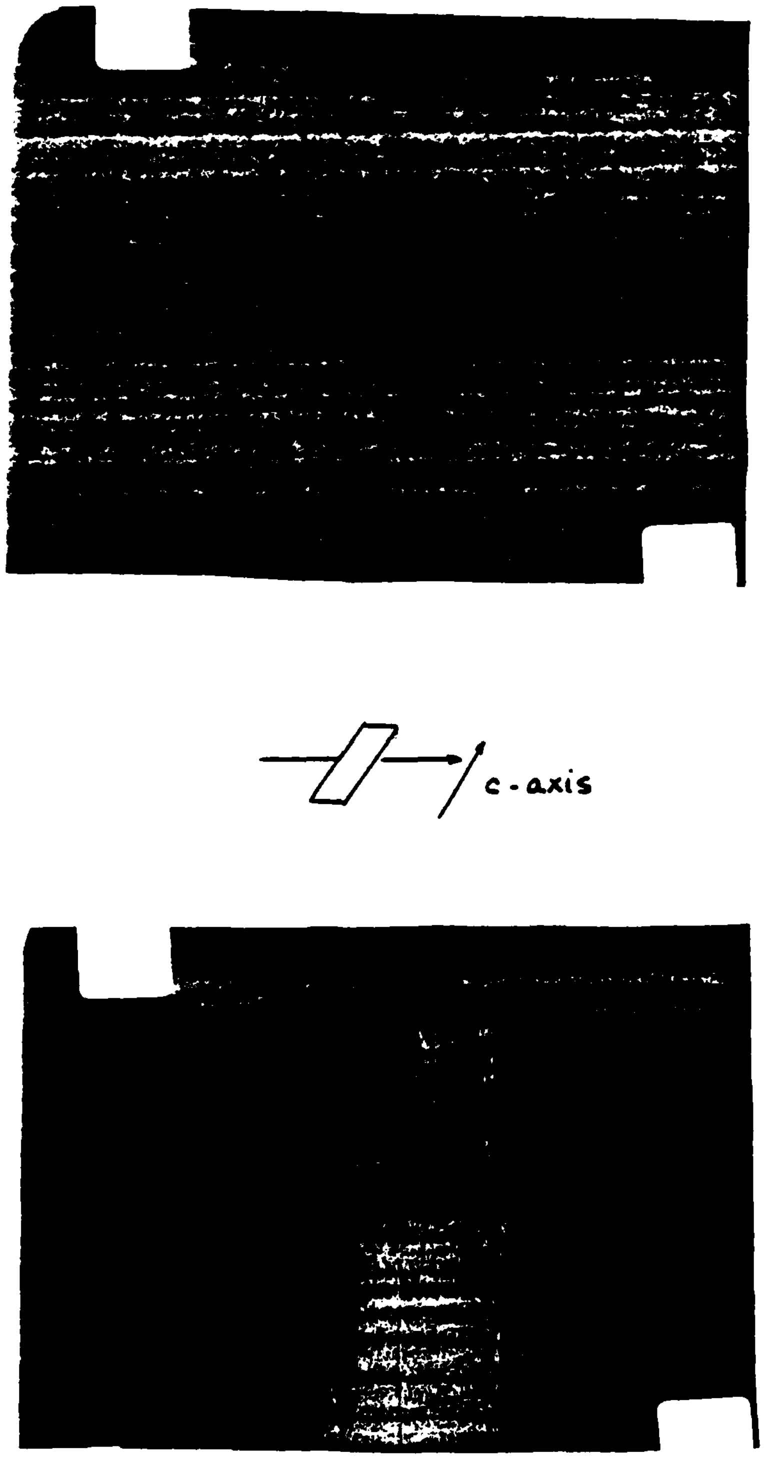


Fig. 15

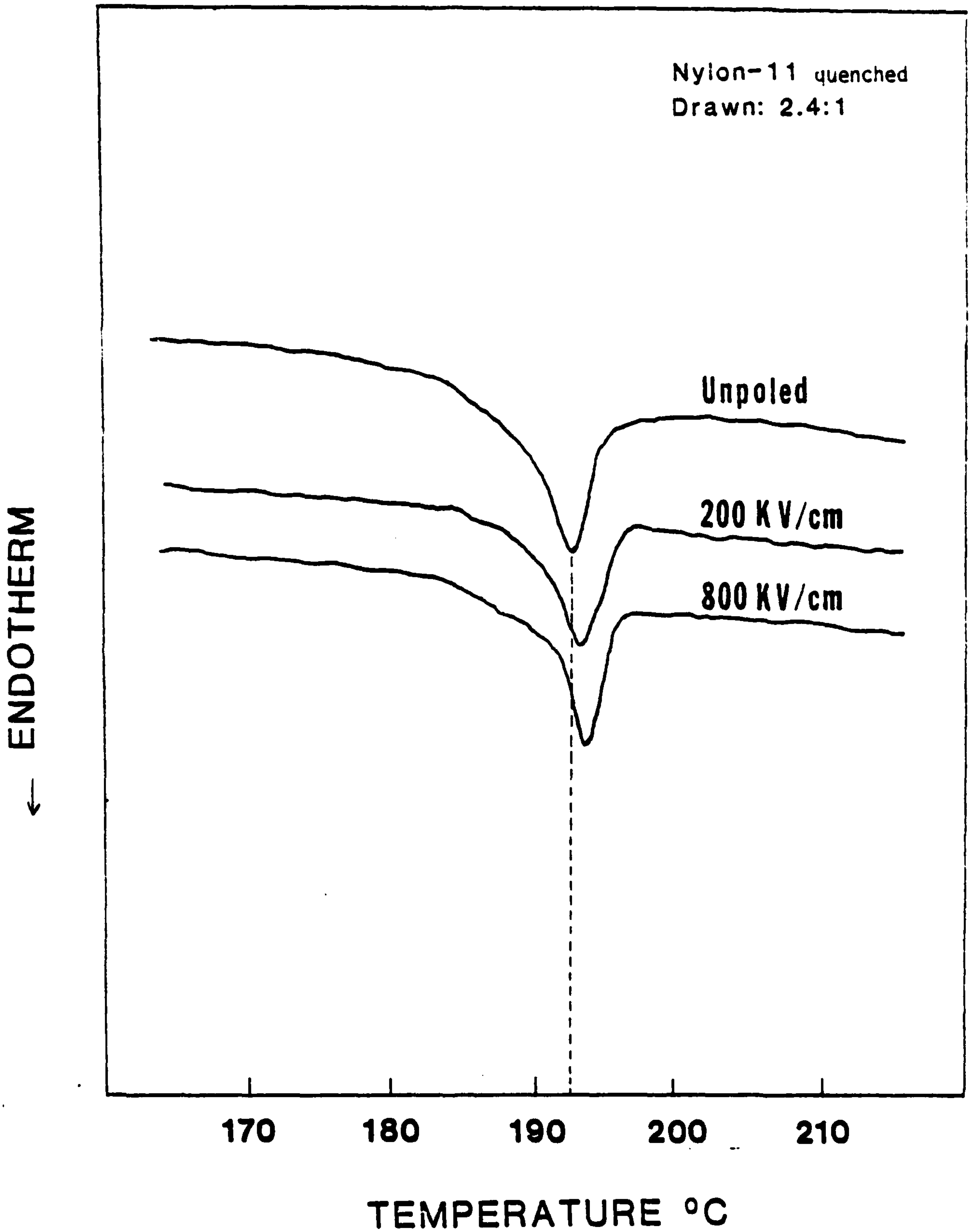

Nylon-11 quenched
Drawn: 2.4:1
Unpoled
200 KV/cm
800 KV/cm
← ENDOTHERM
170
180
190
200
210
TEMPERATURE °C

Fig. 16

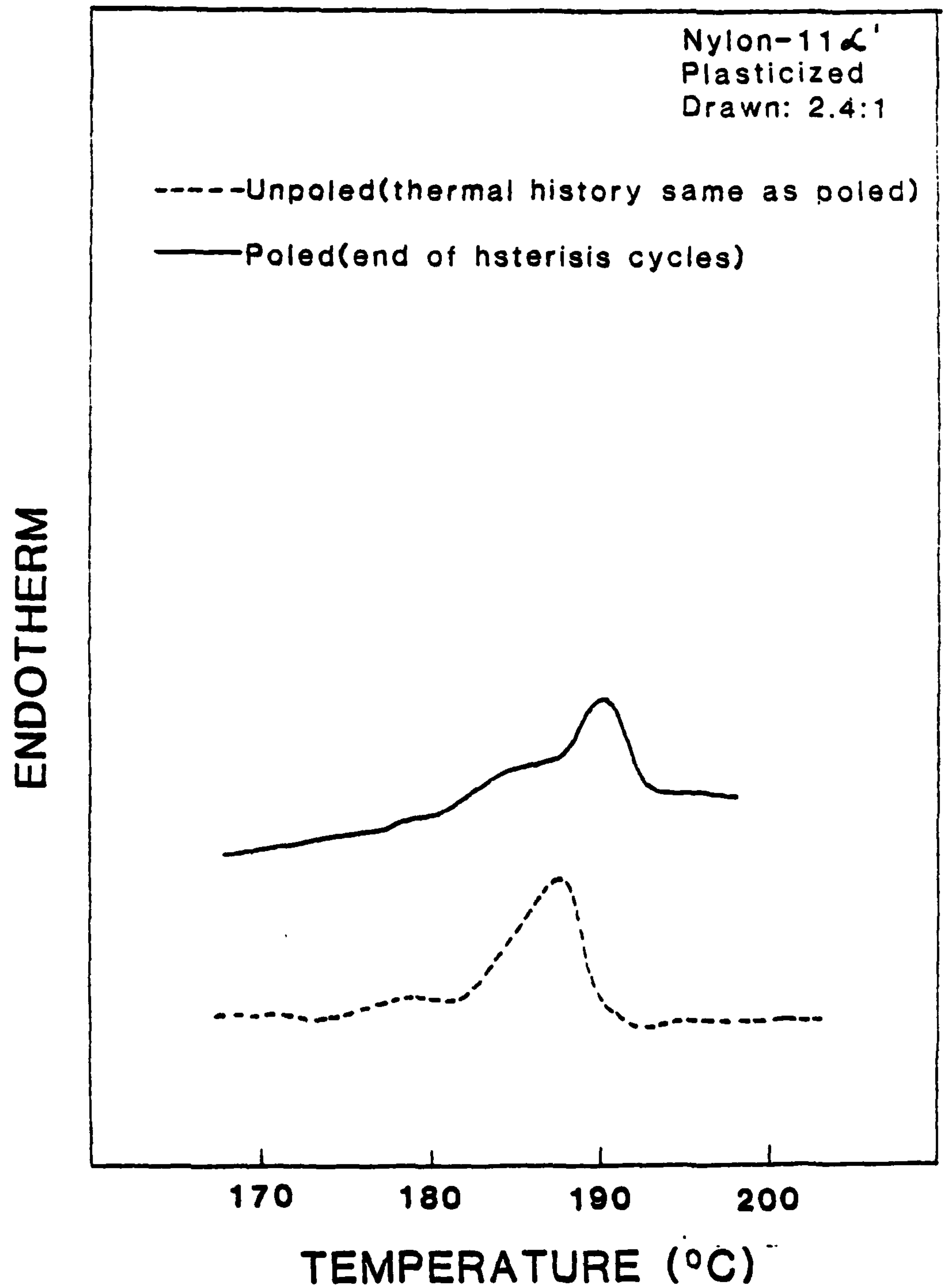

Nylon-11α'
Plasticized
Drawn: 2.4:1
-----Unpoled(thermal history same as poled)
——Poled(end of hsterisis cycles)
ENDOTHERM
170
180
190
200
TEMPERATURE (°C)

Fig. 17

END

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